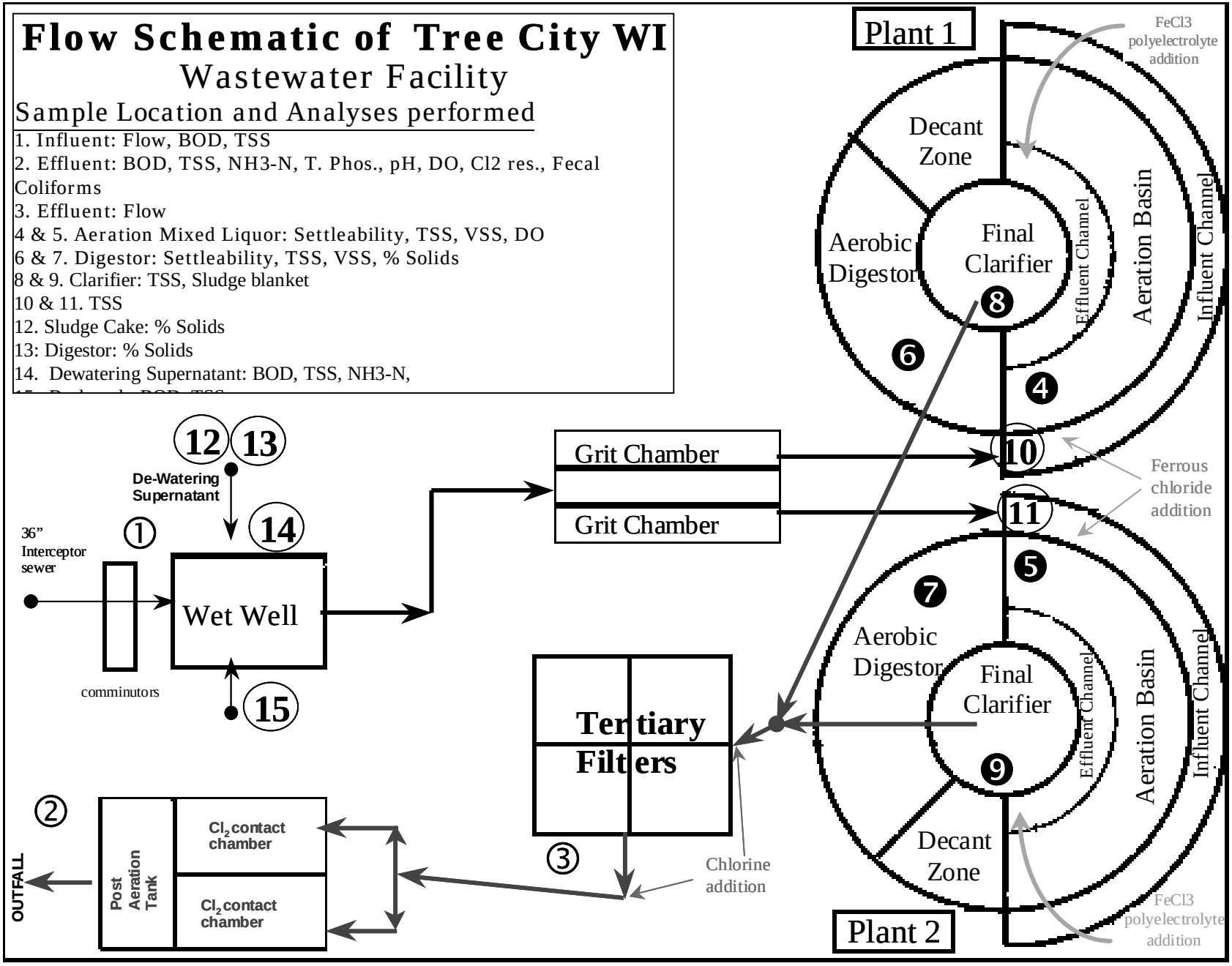
Flow Schematic of Tree City WI

Wastewater Facility

Sample Location and Analyses performed

1. Influent: Flow, BOD, TSS
2. Effluent: BOD, TSS, NH3-N, T. Phos., pH, DO, Cl2 res., Fecal Coliforms
3. Effluent: Flow
4 & 5. Aeration Mixed Liquor: Settleability, TSS, VSS, DO
6 & 7. Digestor: Settleability, TSS, VSS, % Solids
8 & 9. Clarifier: TSS, Sludge blanket
10 & 11. TSS
12. Sludge Cake: % Solids
13: Digestor: % Solids
14. Dewatering Supernatant: BOD, TSS, NH3-N,



1. INTRODUCTION

The laboratory at the Tree City Wastewater Treatment Facility performs analyses necessary both for compliance with requirements specified by the plant's WPDES permit and process control. The lab may also be used to run tests for charge-back of treatment costs to industrial users and other communities. A Grade 3 Operator performs the analysis and reports results as part of other plant operation duties. Quality Assurance (QA) is critical so that decisions resulting from use of the data are based on sound evidence. The purpose of this manual is to document QA activities performed in the lab.

2. MONITORING PROGRAM

Samples are collected to fulfill permit requirements for testing plant influent, effluent, and hauled sludge as well as for industrial and process control monitoring. Wastewater testing requirements are summarized below. Schematic reference numbers correspond to those on the plant schematic (page iv).

Table 1 - WPDES Permit Requirements

SAMPLE LOCATION	SAMPLE TYPE	SCHEMATIC REFERENCE	PARAMETERS TESTED	MONITORING FREQUENCY
Influent	Continuous	1	Flow	Totalized Daily
Influent	24-hr composite (flow proportional)	1	Biochemical Oxygen Demand Total Suspended Solids	Daily
Effluent	24-hr composite (flow proportional)	2	Biochemical Oxygen Demand Total Suspended Solids Ammonia-Nitrogen Total Phosphorus	Daily
Effluent	Grab	2	Dissolved Oxygen pH Chlorine Residual #	Daily
Effluent	Grab	2	Fecal Coliform #	Twice weekly

- Monitoring only required during the period from May 1 to September 30 in any given year

The permit also requires that a sludge characteristic report be submitted annually for quarterly analyses. Sludge analyses for non-routine parameters are performed by a certified commercial laboratory.

INDUSTRIAL MONITORING

For billing purposes and operational considerations, all major industries are simultaneously monitored twice each year. Sampling is flow-proportional over a three day period. Routine analyses are BOD, suspended solids, phosphorus and continuous pH. The laboratory is registered with the Wisconsin Laboratory Certification and Registration Program to perform BOD, ammonia, TP, and TSS. Sulfates, chlorides, metals, and oil and grease are sent to a certified commercial laboratory; however, the plant's lab may be used for testing needed to locate a source of wastewater contributing to a plant upset. Samples sent to commercial labs are preserved within 15 minutes of collection.

Table 2 - Process Control Monitoring

SAMPLE LOCATION	SAMPLE TYPE	SCHEMATIC REFERENCE	PARAMETERS TESTED	MONITORING FREQUENCY
Aeration Tank	Outlet grab	4 & 5	Settleability (30 min.) Total Suspended Solids Volatile Solids	Daily
Aeration Tank	Contents in-place	4 & 5	Dissolved Oxygen	Continuous
Solids Concentrator	Product-grab	12 & 13	Percent solids	As needed
Solids Concentrator	Decant-grab	14	Biochemical Oxygen Demand Total Suspended Solids Ammonia-Nitrogen	As needed
Digester Contents	Grab	6 & 7	Settleability (30 min.) Percent solids Total Suspended Solids Volatile Suspended Solids	Daily
Clarifier	Grab	8 & 9	Blanket Depth Total Suspended Solids	Daily
Return Sludge	Grab	10 & 11	Total Suspended Solids	Daily
Filter Backwash	Grab	15	Biochemical Oxygen Demand Total Suspended Solids	As needed

3. SAMPLING

A flow-proportional automatic sampler is used to sample from the influent channel just upstream of the raw wet-well. The effluent at Tree City WWTF is sampled after all treatment units and after chlorination and subsequent dechlorination at one sample location just prior to discharge. Some other treatment plants may have been designed in a way which would allow effluent samples to be composited and collected before the disinfection step so that seeding BOD samples may not be necessary. These samplers receive signals from the influent flow meter so that sampling is done in a flow proportional mode. Samplers have refrigeration units that maintain sample temperatures at 4 °C.

The operator collects samples from the automatic samplers at approximately 7:00 a.m. by replacing the filled sample containers (polyethylene) with clean containers and transporting samples directly to the lab. Flow meter readings and the temperature of the automatic sampler are recorded when samples are collected. Samples are allowed to stand at room temperature while calibration checks are performed. Analyses begin at approximately 8:00 a.m.

Grab samples are collected during the mid-afternoon by the operator in plastic bottles for direct transport to the lab. Samples for chlorine residual and pH are tested immediately (within 15 minutes). Samples for fecal coliform are collected in a sterilized glass jar containing a drop of 10% sodium thiosulfate solution. Testing is initiated shortly thereafter.

Processed sludge for the required quarterly Sludge Characteristic Report is collected by compositing hourly grab samples. These are collected during a normal 7-hour run of the solids processing unit. Sludge samples collected for the analysis of percent solids, pH, and nutrients (ammonia-nitrogen, Total Kjeldahl Nitrogen [TKN], total phosphorus) are exempted from the requirement that they be performed by a registered/certified laboratory as outlined in NR 219.07. Samples for metals and other non-routine analyses are composited into a polyethylene container provided by the commercial lab, refrigerated, and transported to the commercial lab the following day. Samples are shipped by overnight express in coolers with ice when temperature presentation is required. Typical analyses required include:

arsenic, cadmium, chromium, copper, lead, mercury, nickel and zinc.

All sludge results must be reported on a dry weight basis. If data are received "as is" or calculated based on "wet weight", they can be converted to dry weight using the following formula:

$$\text{Analyte (mg/kg dry weight)} = \frac{\text{mg/kg wet weight}}{(\%\ \text{solids}/100)}$$

Example: Zinc = 2.5 mg/kg dry , 80% solids = $\frac{2.5}{(80/100)}$ = 3.125 mg/kg dry weight

On occasion, the DNR region may require the analysis of additional parameters from the list of Conventional Priority Pollutants. These analyses are also performed by a commercial laboratory, which will, in many cases, supply the sample bottles (containing required preservatives) and sampling guidance.

Sample bottles for analyses performed at the wastewater facility are permanently labeled for their appropriate use. Any departure from standard sampling protocol is noted on appropriate bench sheets. Care is taken to ensure that samples are well mixed prior to aliquot withdrawal. All sample bottles are washed with detergent after each use. Bottles for phosphorus samples are washed with a non-phosphate detergent and rinsed with 1:1 hydrochloric acid. Bottles for fecal coliforms are sterilized before use.

Sample handling/preservation requirements for wastewaters are in Table 3. Sample handling and preservation methods required by state and federal laws must be followed (see NR 219, Table F).

SAMPLER CLEANING

The sampler should be cleaned twice monthly using the following procedure:

1. Pump HOT tap water through the tubing: run the sampler for at least two (2) minutes.

2. Rinse the tubing: pumping a 20% hydrochloric acid or a 20% nitric acid solution for an additional two minutes. This acid rinse is reused up to four (4) times before it is discarded. Safety precautions are used in the handling and disposal of these acid solutions (wear safety gloves and glasses).

3. Re-rinse the tubing again: pump HOT tap water through it for at least two (2) minutes.

4. Rinse the tubing by pumping distilled water through the system for at least one (1) minute. After one minute, stop the pump and allow the water to stand in the hose for an additional minute. After this minute, continue pumping the water for one (1) final minute. The distilled rinse is NOT re-circulated. To ensure the bottles are free of materials that may contribute to the BOD, a weak solution of household bleach (50 mL per 2 L of deionized water) can be used as a final step in the cleaning procedure. *The final rinse, however, must be sufficient to eliminate any traces of the bleach, which may kill natural seed organisms, leading to low bias in BOD values.*

The sampler itself is subjected to a good general washing. Clean the pump, sample container(s), and any internal sampler parts which come into contact with the wastewater. First wash the sampler parts with tap water. Next, vigorously scrub all parts with hot tap water and a non-Phosphorus detergent. Triple rinse (i.e. rinse and drain, rinse and drain, rinse and drain) these items with tap water after washing. Finally, follow this process up with three (3) rinses using distilled water.

TABLE 3 - SAMPLE HANDLING TABLE
(This table may be customized to reflect the lab's actual practices)

PARAMETER	SAMPLE TYPE	PRESERVATION	CONTAINER	@MAXIMUM HOLDING TIME	*ANALYTICAL METHOD
Biochemical Oxygen Demand	24-hr composite [flow proportional]	Cool, 4°C	Polyethylene	24 hours	5210 B
Total Suspended Solids	24-hr composite [flow proportional]	Cool, 4°C	Polyethylene	7 days	2540 D
Ammonia-Nitrogen	24-hr composite [flow proportional]	Cool, 4°C; H_2SO_4 to pH <2	Polyethylene	28 days	4500-NH_3 F
Total Phosphorus	24-hr composite [flow proportional]	Cool, 4°C; H_2SO_4 to pH <2	Polyethylene	28 days	4500-P B(5) & 4500-P E
pH	Grab	None	Polyethylene	Analyze immediately	4500-H^+ B
Chlorine Residual	Grab	None	Polyethylene	Analyze immediately	4500-CL G
Fecal Coliform Bacteria	Grab	Cool, 4°C; 0.008% $Na_2S_2O_3$	Polyethylene	6 hours	9222 D

NOTES: @ From time of completed sampling
* Standard Methods for the Examination of Water and Wastewater, American Public Health Association, 18th Edition, 1992.

4. ANALYSES

Accepted test methods are used for all analyses. Refer to Table 3 for a summary of specific methods used. Refer to the appendix for a detailed description of individual methods.

BOD

In a nutshell, BOD gives a measure on the impact of a waste(water) on the oxygen content of a receiving system(stream/river/lake). Wastes are broken down by microbial organisms (frequently referred to as "bugs"), and the bugs, in turn, require oxygen for this monumental effort. Thus, in order for this test to "work", you need (1) a food source, (2) a nice population of bugs, (3) available oxygen to drive the bugs, and (4) a system which provides a hospitable environment for the bugs.

A series of dilutions with nutrient-laden, buffered dilution water is performed on each sample. Samples may also be seeded with a population of microorganisms as necessary. An initial measurement of dissolved oxygen is obtained, and then again following a five-day incubation period at 20 ± 1 °C. The extent to which oxygen is depleted is used to calculate BOD. In that the BOD test is actually a bioassay, it is critical that documentation of conformance with all method parameters is maintained.

AMMONIA

All of the approved procedures for the determination of ammonia require a preliminary distillation step. The requirement to perform this procedure, outlined in Standard Methods 4500-NH_3 B can be waived under certain circumstances. Note 8 under Table B of NR 219indicates that, "*Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.*"

The State Laboratory of Hygiene performed a detailed comparison study of domestic wastewater samples analyzed for ammonia both with and without preliminary distillation. The results of this study are available through the Laboratory Certification and Registration Program; each facility should maintain a copy of this paper in their files. If your facility is processing anything other than domestic wastewater (i.e., paper mill effluent, cheese wastes, industrial pre-treatment), you may be required to either (1) distill all samples, or (2) perform an individual comparability study.

Distillation is performed at a basic pH (~ 9.5). Distillate is collected in boric acid if followed by Nesslerization methods, in indicating boric acid if followed by titration methods, in sulfuric acid (1N) if followed by phenate methods, or in dilute sulfuric acid (0.04N) if followed by electrode methods.

The most common method used is the ion-selective electrode technique. Raising the sample (or distillate) pH above 11 with a strong base (10N NaOH) causes conversion of dissolved NH_3 and any NH_4^+ in the sample to NH_3. Diffusion of gaseous NH_3 through the hydrophobic membrane causes changes in the electrode's internal solution (NH_4Cl) which in turn registers as a change in millivolt response on the meter. The EPA is moving towards removing approval for the Nessler technique due to the toxicity of reagents used.

PHOSPHORUS

Because of the prevalence of phosphorus in the environment (although many detergents have become significantly more environmentally friendly over time), the phosphorus determination is the easiest test to introduce analyst contamination. All glassware must be scrupulously cleaned with non-phosphorus detergents, then acid washed and rinsed. Phosphorus is present in our skin oils also, so be sure that fingers do not come in contact with inside of sample beakers, or other glassware. It is a generally a good idea to dedicate glassware specifically for phosphorus determinations.

All samples are subject to an initial digestion using either a hotplate or an autoclave. The digestion step oxidizes all organically bound phosphorus into orthophosphate. Ammonium molybdate and potassium antimonyl tartrate react with orthophosphate under acidic conditions to produce phosphomolybdic acid. This compound is in turn reduced by ascorbic acid to molybdenum blue, which is easily recognized by its intensely blue color. Chapter NR 219 references three procedures approved for phosphorus determinations in wastewater, each of which employs ascorbic acid and the formation of a blue color. Absorbance is typically measured at 880 or 650 nm.

Several other methods are listed in Standard Methods, however, they are not currently approved for use in wastewater analyses:

- a Stannous chloride procedure, which also produces a **blue** color, but absorbance is measured at **690 nm**,
- a Vanadomolybdophosphoric acid procedure which produces a **yellow** color measured at **400-490 nm**,

NOTE: Your laboratory must change its phosphorus procedure if (1) you are measuring absorbance at less than 650 nm, (2) the color of the solution you are measuring is yellow, or (3) if you are using stannous chloride in the color-producing step.

TOTAL SUSPENDED SOLIDS (TSS)

This procedure quantifies the mass of materials in a water system that are trapped on a glass fiber filter of pore size approximately 1.0 - 1.5 microns. The most critical aspects of this method are to adequately dry the sample at the correct temperature, and to capture an appropriate amount of residue.